

SELECTION AND CHARACTERIZATION OF LOW-RANK COAL SAMPLES

S.A. Benson, D.R. Kleesattel, and H.H. Schobert

University of North Dakota
Energy Research Center
Box 8213, University Station
Grand Forks, North Dakota 58202

INTRODUCTION

The collection of low-rank coal samples at the University of North Dakota Energy Research Center (UNDERC) includes coals from the major lignite and sub-bituminous coal regions of the United States. Primarily, the coals were collected to provide fresh, homogeneous, and well characterized samples for projects in coal science research at UNDERC. In the past, research has been performed on coal in which no information was reported or available on how the samples were prepared and stored, or on the geography and geology of the coal seam. Richard Neavel (1) pointed out that work on "poorly selected, poorly collected, poorly prepared, poorly preserved and poorly characterized coal samples will only lead us into further confusion." It is important that coal science research be performed with carefully collected, prepared, and documented samples so that the resulting data on coal properties, composition, and reactivity will be meaningful to other researchers.

In the past several years a great deal of interest has surfaced dealing with sample banks. The American Physical Society (2) identified the need for a sample bank of well-selected, characterized and preserved coal samples the lack of which has been a major obstruction in the past to advancement of coal research. A Coal Sample Bank Workshop was held in March 1981 sponsored by the Gas Research Institute and the U.S. Department of Energy to identify the need and problems associated with setting up a National Coal Sample Bank (3). It is our hope that the information presented in this paper will be of use to researchers interested in establishing a sample bank or interested in incorporating low-rank coal samples in existing sample banks. The motivation for such work at UNDERC is the need for this type of research to be accomplished for low-rank coals.

Channel samples of low-rank coals have been collected from various mines in North Dakota, Montana, Colorado, New Mexico, and Texas. The samples were carefully collected, homogenized, and stored in an inert atmosphere. The coals were prepared in several different sizes from -1/2 inch to -60 mesh and quantities from 15 kilograms to 250 grams. At UNDERC the coals will be used to study various coal features such as the distribution of inorganics, organic structure, ash and slag chemistry, physical and mechanical properties, and pyrolysis and extraction behavior. In addition, trace element analysis, proximate, ultimate, heat content, ash fusion, and ash analysis will be performed.

SAMPLE SELECTION AND COLLECTION PROCEDURES

The selection of coals was based either on some desired unique characteristics such as sodium content, or slag viscosity, or on production tonnage. The goal is eventually to have a set of coals available which represent a wide range of coal properties characteristic of low-rank coals. The selection and preparation of coals will be an ongoing project at UNDERC for at least two more years and the total suite of samples will extend beyond the coals collected to date. The coal ranks to be included range from low-grade lignite to high-rank subbituminous.

When a mine has been chosen for sampling, a pit within the surface mine is selected, based either on some unique characteristic or on ready accessibility to the pit. A "typical section" is selected for sampling which is free of faults, slumping, or evidence of groundwater flow. The face of the seam or seams to be sampled is cleaned to expose fresh nonweathered coal free from extraneous mineral matter which may have fallen from the overburden. The coal seam is measured and a megascopic description is made of the coal and associated sediments. A channel sample is collected to provide approximately 100 kilograms of coal. In some cases, one-kilogram samples are also collected at various intervals throughout the seam, including overburden, interburden (if two seams are sampled) and underclay, to study the within-seam variability. All samples are sealed in plastic bags and transported to UNDERC for preparation. A list of samples collected through November 1983 is in Table 1.

TABLE 1
LOW-RANK COALS COLLECTED

<u>Mine</u>	<u>Rank</u>	<u>State</u>	<u>Region</u>
Abasloka (Sarpy Creek)	Subbituminous	Montana	Powder River
Indian Head	Lignite	North Dakota	Fort Union
Beulah (High-Na)	Lignite	North Dakota	Fort Union
Beulah (Low-Na)	Lignite	North Dakota	Fort Union
Gascoyne (Red Pit)	Lignite	North Dakota	Fort Union
Gascoyne (Blue Pit)	Lignite	North Dakota	Fort Union
Velva	Lignite	North Dakota	Fort Union
Spring Creek	Subbituminous	Montana	Powder River
Falkirk	Lignite	North Dakota	Fort Union
Glenharold	Lignite	North Dakota	Fort Union
Colorado Coal Co.	Subbituminous	Colorado	Green River
Navajo	Subbituminous	New Mexico	San Juan
San Miguel	Lignite	Texas	Gulf Coast
Martin Lake	Lignite	Texas	Gulf Coast
Savage	Lignite	Montana	Fort Union
Center	Lignite	North Dakota	Fort Union

The channel sample is crushed, split, and stored in plastic or glass containers under an argon atmosphere. The crushing is done using a hammer mill. A diagram which illustrates the procedure used for preparing the coals is shown in Figure 1. The final splitting of the -60 mesh coal is done by a rotary riffle in an argon-purged glove box. These samples are stored in plastic or glass containers which are in turn sealed in a plastic pouch. The -8 mesh coals are stored in plastic containers and purged with argon.

The samples collected at various locations within the seam are prepared for selected characterization techniques, such as petrography of separated lithotypes; x-ray diffraction of the overburden; interburden; underclay; and trace element analysis. This type of work is being done to examine the variability of organic and inorganic constituents within the seam and to understand interrelationships between coal properties.

RESULTS AND DISCUSSION

The geologic and geographic description of the sampling area along with the analytical determinations will be available for all samples. The types of analytical results being obtained include proximate, ultimate, heat content, ash fusion, ash analysis, and trace element analysis. As an example, the results obtained for Indian Head lignite will be summarized here. Table 2 summarizes the geographic and geologic description of the Indian Head Mine. The results of the analytical determinations for the traditional ultimate, proximate, heat content and ash analysis are summarized in Table 3. The trace element determinations performed by neutron activation analysis (NAA) are listed in Table 4. The NAA analyses were made by North Carolina State University (4).

TABLE 2
GEOGRAPHIC AND GEOLOGIC DESCRIPTION
OF INDIAN HEAD MINE

County	Mercer
State	North Dakota
Town	Zap
Basin	Williston
Group	Fort Union
Formation	Sentinel Butte
Bed	Beulah-Zap
Age	Paleocene
Grand Forks Number	83-0008
Sampling Date	11/83

The information shown in Tables 2-4 is steadily supplemented by characterization data from several research groups within the UNDERC Coal Science Division, which include Ash and Slag Chemistry, Organic Chemistry, Distribution of Inorganics and Geochemistry, Physical Properties and Moisture, and Coal Reactivity. Samples of lithotypes have been separated for characterization by many of the above techniques to supplement the "whole coal" data. Petrography is also being performed on the homogenized channel sample and the separated lithotypes.

SUMMARY AND CONCLUSIONS

The low-rank coals have been collected to obtain coals which represent a broad range of low-rank coal characteristics. These coals are carefully collected, prepared, and stored under an argon atmosphere. These samples are not comparable to those in a premium sample bank, because they are stored at room temperature and are not collected in large reserve quantities. All the data generated will be entered in a computer system and can be readily accessed. The results of studies on these coals will provide insight into the interrelationships of various coal properties, both with each other and with coal-specific process responses.

TABLE 3
RESULTS OF ANALYSES FOR INDIAN HEAD LIGNITE

<u>Ultimate Analysis</u>	<u>Wt % (as received)</u>
C	44.08
H	6.36
N	0.64
S	0.38
<u>Proximate</u>	
Moisture	34.0
Volatile Matter	27.4
Fixed Carbon	33.8
Ash	4.8
<u>Heat Content</u>	7329 Btu/lb
<u>Ash Analysis</u>	<u>Wt % of ash</u>
SiO ₂	21.1
Al ₂ O ₃	11.9
Fe ₂ O ₃	8.5
TiO ₂	1.0
P ₂ O ₅	0.5
CaO	20.9
MgO	6.5
Na ₂ O	11.9
K ₂ O	0.1
SO ₃	15.6
TOTAL	98.1

Mineralogy of coal by x-ray diffraction of low-temperature ash -
quartz
kaolinite
pyrite

Carboxylic acid group content 2.48 meq/gram.

ACKNOWLEDGMENTS

This work is funded by the U.S. Department of Energy, Office of Fossil Energy, under Cooperative Agreement DE-FC01-83FE60181.

TABLE 4

NEUTRON ACTIVATION ANALYSIS OF INDIAN HEAD LIGNITE FROM NORTH DAKOTA

<u>Element</u>	<u>Concentration, ppm</u>
Titanium	110 \pm 20%
Iodine	<3.0
Manganese	4.6 \pm 5%
Magnesium	410 \pm 15%
Copper	<25.0
Vanadium	3.00 \pm 2%
Chlorine	16 \pm 20%
Aluminum	3020 \pm 1%
Samarium	0.4 \pm 2%
Uranium	0.3 \pm 10%
Lanthanum	4.7 \pm 1%
Cadmium	<1.0
Gold	<0.001
Arsenic	5 \pm 1%
Antimony	0.2 \pm 1%
Bromine	1.1 \pm 1%
Sodium	6220 \pm 1%
Potassium	<500.0
Cerium	7.80 \pm 10%
Calcium	3760 \pm 10%
Selenium	0.50 \pm 10%
Thorium	1 \pm 1%
Chromium	2 \pm 15%
Europium	0.09 \pm 10%
Ytterbium	0.4 \pm 20%
Barium	520 \pm 5%
Cesium	0.1 \pm 10%
Silver	<0.08
Nickel	<25.0
Scandium	1.3 \pm 1%
Rubidium	<5.0
Iron	3570 \pm 3%
Zinc	6.4 \pm 20%
Cobalt	1.60 \pm 1%
Silicon	<35,000.0
Molybdenum	<10.0

REFERENCES

1. Neavel, R.C. "Coal Structure and Coal Science: Overviews and Recommendations", 178th National Meeting of the ACS, Honolulu, Hawaii, Fuel Division, Preprints, Vol. 24, No. 1, p. 73, 1979.
2. Reviews of Modern Physics, Vol. 53, No.4, Part II, October 1981.
3. Coal Sample Bank Workshop, Atlanta, Georgia, March 1981, sponsored by GRI/DOE.
4. Weaver, J.N. "Analytical Methods for Coal and Coal Products," Karr, C., Ed., Academic Press: New York, Vol. 1, pp. 377-401, 1978.

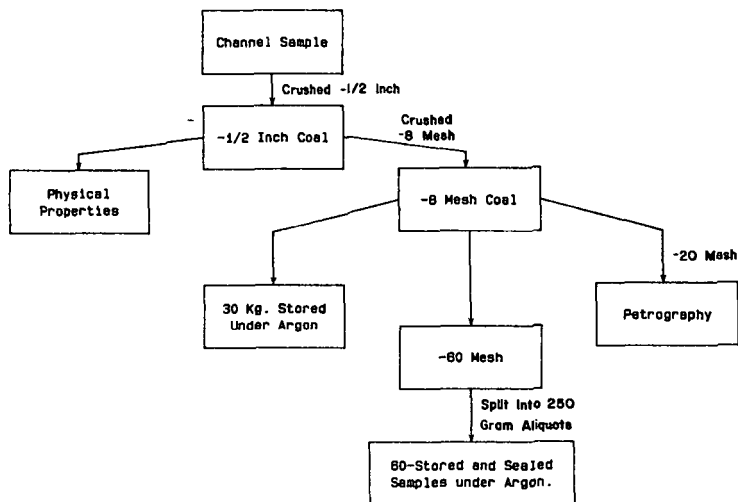


FIGURE 1. Schematic of sample preparation procedure for channel sample.